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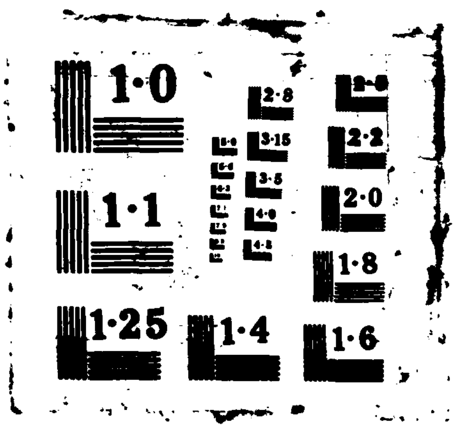
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During the six month period October 1, 1985--March 31, 1986, efforts on the Program "Synthetic Metals from Intercalated Graphite" were largely focused on studies of magnetic graphite intercalation compounds. Magnetoresistance measurements of the magnetic field-induced phase transitions at high magnetic fields in the model donor compound Europium intercalation compounds were interpreted using a Monte Carlo simulation to gain insight into magnetic interactions between the graphite pi electrons and the intercalant f-electrons.

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Contents

1.2 Statement of Work

Statement of work in AFOSR contract #F49620-83-C-0011 on Structure-Property Relationships in Intercalated Graphite.

- Derive techniques for improved methods for the preparation and characterization of specific graphite intercalation compounds.
- Synthesize new intercalated systems and study their structure.
- Study in-plane structure and phase transitions in the intercalate layers with electron diffraction, lattice fringing, real space electron microscope imaging, and high resolution x-ray scattering.
- Deduce structural phase diagrams for specific graphite intercalation compounds.
- Investigate in detail commensurate-incommensurate phase transitions.
- Study lattice modes by infrared and Raman spectroscopy, and inelastic neutron scattering.
- Derive models for the phonon dispersion relations throughout the Brillouin zone, and apply these models to interpret lattice mode studies.
- Model the electronic dispersion relations and apply these models to interpret the experimental results relevant to the electronic properties.
- Measure and model thermal transport phenomena in intercalated graphite.
- Measure the temperature and field dependence of the magnetic susceptibility and heat capacity of magnetic graphite intercalation compounds and to construct magnetic phase diagrams for these systems.
- Study the superconductivity of specific graphite intercalation compounds.

2 Current Status of Research Effort

A summary of the current status of the research effort on the "Synthetic Metals from Intercalated Graphite" is presented in terms of the progress made during the period October 1, 1985 to March 30, 1986. In presenting the summary, we refer by number (#n) to the publications for the six month period October 1, 1985 to March 30, 1986 following the publication numbers which are listed in section 3.1.

2.1 Low Dimensional Magnetism in Magnetic Graphite Intercalation Compounds

In the funding period October 1, 1985 – March 31, 1986, increasing emphasis has been given to studying the novel anisotropic properties of magnetic intercalation compounds and their use as model materials for the physical realization of current theories of two–dimensional magnetism. The Ph.D. thesis work by S.T. Chen completed in November 1985, achieved major advances in our understanding of both acceptor and donor magnetic systems. Though most of his work was directed toward magnetically ordered systems and stage 1 compounds, the Ph.D. Thesis by Chen laid the foundation for quantitative studies of the two–dimensional aspects of the problem. James Nicholls joined the project in June 1985, and his Ph.D. thesis will focus on the much more difficult two–dimensional aspects of the problem, both experimentally and theoretically. Hector Jiménez-Gonzaléz, a young fellowship student who has not yet passed his qualifying exams, is looking into the synthesis of new kinds of magnetic graphite intercalation compounds with particular emphasis on magnetic donor compounds. Djalma Domingues, another fellowship student who very recently joined our research group (March 1986) is assisting with the preparation of magnetic GIC samples. James Speck, an NSF fellowship student who has just passed the doctoral qualifying exams, has been successful with preliminary high resolution transmission electron microscopy studies in $MnCl_2$–GICs, and it is expected that a major part of his Ph.D. thesis will be devoted to understanding the microstructure and intercalate island formation which dominate the magnetic properties of magnetic acceptor GICs. N.C. Yeh, who has been working primarily on charge transfer phenomena in the ternary KH_x–GIC system, is now devoting part of her efforts to susceptibility and magnetoresistance measurements in the antiferromagnetic $MnCl_2$–GIC compounds, and she has already obtained some interesting results on this acceptor system. We expect this restructuring of the overall research program on graphite intercalation compounds, will be yielding many new results on magnetic graphite intercalation compounds in the next six month period. We now describe progress that has been made in the October 1, 1985 – March 31, 1986 period.

2.1.1 Magnetoresistivity and Monte Carlo Studies of Magnetic Phase Transitions in C_6Eu

In the first stage donor compound C_6Eu, a number of unusual magnetic phase transitions occur at high magnetic fields.(# 1) We have found that these magnetic phase transitions are sensitively probed by high field magnetoresistance measurements, utilizing the unique facilities at the Francis Bitter National Magnet Laboratory. To explore the magnetic phase diagram of this prototype magnetic donor compound, the high field magnetoresistivity $\rho(H)$ of the antiferromagnetic first stage graphite intercalation compound C_6Eu has been measured with both $\vec{H} \perp \hat{c}$ and $\vec{H} \parallel \hat{c}$. Both longitudinal ($\vec{J} \parallel \vec{H}$) magnetoresistivity $\rho_\ell(H_\perp)$ and transverse ($\vec{J} \perp \vec{H}$) magnetoresistivity $\rho_t(H_\perp)$ with $\vec{H} \perp \hat{c}$ show distinct changes across the magnetic phase boundaries which occur at fields of 1.5T, 8T, 15T and 21.5T at a temperature T = 4.2 K. The phase transition at H=15T was not observed previously by the pulsed magnetization measurements. A Monte Carlo simulation based on the Hamiltonian of Sakakibara and Date was carried out for the C_6Eu system to identify the spin configuration for each of the magnetic phases. The 15T phase transition reported for the first time in this work, is explained as a transition from a "canted" to a "fan" state. The transverse magnetoresistivity $\rho_t(H_\parallel)$ with $\vec{H} \parallel \hat{c}$ shows a clear anomaly at the field corresponding to the onset of the transition to the spin aligned paramagnetic state. A magnetic phase diagram has been accurately determined based on the results of the magnetoresistivity measurements for both $\vec{H} \perp \hat{c}$ and $\vec{H} \parallel \hat{c}$. The various spin configurations in the phase diagram for $\vec{H} \perp \hat{c}$ are identified and the parameters of the Hamiltonian are determined using the results of the Monte Carlo simulation.

2.1.2 Electrical Resistivity, Magnetoresistance and Magnon Drag Effect in C_6Eu

Experimentally, discontinuities in the resistivity as a function of magnetic field are used to sensitively identify magnetic phase transitions in the prototype donor compound, stage 1, C_6Eu (# 1). A calculation of the magneto-resistance for C_6Eu has been carried out (# 4) to understand the sign and the magnitude of the discontinuity in the magnetoresistance resulting from these magnetic phase transitions. The spin arrangements for each of the magnetic phases has been determined independently using a Monte Carlo simulation technique (# 1).

The localized f-electron system with spin S = 7/2 in C_6Eu interacts strongly with mobile π-electrons via the π-f exchange interaction. The spin system exhibits four different spin configurations as the varying magnetic field applied along the basal plane is increased to the 25 tesla range. As a consequence, the in-plane resistivity shows interesting dependences on magnetic field and temperature associated with each spin configuration. The electrical resistivity and magnetoresistance are calculated considering the spin fluctuation scattering due to the π-f exchange interaction. Detailed calculations are performed in the ferrimagnetic spin configuration. Though the theory provides a qualitative explanation for the observed field dependence of the magnetoresistance, the calculated temperature dependence is stronger than the observed one. This discrepancy is removed by introducing a backward momentum flow due to a non-thermal distribution in the magnon system. Since the π-f exchange interaction is strong, this magnon drag effect enhances the π-electron conductivity, especially at high temperatures. By including the magnon drag effect, better agreement is obtained with the measured temperature dependence of the magnetoresistance. The magnetoresistance in the ferromagnetic configuration is also calculated. In this region, the effect of the orbital motion on the resistivity is important especially at low temperatures ($T < 10K$), while the magnon drag effect is expected to be important at high temperatures.

2.1.3 Electronic and Magnetic Properties of $MnCl_2$-GICs

$MnCl_2$-GICs are of interest as a possible example of a quasi-2D Heisenberg or XY antiferromagnetic system (# 9). In its pristine form, $MnCl_2$ has antiferromagnetic coupling for Mn ions within a Mn plane and a weaker ferromagnetic coupling to Mn ions out of the plane. The in-plane Mn ions form a triangular lattice with a lattice constant b = 3.69 Å. With the intercalation of $MnCl_2$ into graphite, the in-plane structure is maintained, but the adjacent Mn planes are further separated by graphite layers, thus greatly reducing the interplanar ferromagnetic coupling. The Néel temperatures (T_N) of stage-1 and stage-2 $MnCl_2$-GICs are at 1.28 K and 1.10 K, respectively. Above these temperatures, the Mn ions exhibit paramagnetic properties, but as the temperature decreases below T_N, the system undergoes a phase transition to an antiferromagnetic state. To understand the possible effects of the localized spins on the transport properties of the graphite conduction π-electrons as well as the electronic properties of this system, magnetoresistance measurements were carried out on stage 1 $MnCl_2$-GIC samples prepared in our laboratory. The measurements were made with magnetic fields up to ~20 tesla.

The effects of the localized spins on the transport properties of the graphite π-electrons as well as the electronic properties of $MnCl_2$-GICs have been studied by measuring their magnetoresistance and temperature-dependent resistivity. For temperatures above the N/'eel temperature (T_N), a negative magnetoresistance was observed for fields $0 \leq H \leq 2$ tesla. This negative magnetoresistance is attributed to the spin-disorder scattering of π-electrons from the localized d-electrons surrounding the Mn ions. An anisotropic magnetoresistance was also observed as qualitatively different behavior for $\vec{H} \parallel \hat{c}$ and $\vec{H} \perp \hat{c}$, and this effect may be explained by the nearly 2-dimensional properties of this system. Magnetoresistance and temperature-dependent resistivity experiments are planned for the low temperature range $T \leq T_N$ to obtain better understanding of the 2D antiferromagnetic spin effects on the transport properties of the system. In addition, similar experiments on other transition metal-chloride GICs will be carried out to look for possible universal effects in the transport properties in these related magnetic systems.

One important finding has been the observation of a negative magnetoresistance effect. Even more interesting is the anisotropy of the negative magnetoresistance effect. The zero field temperature dependent resistivity was also measured from 4.2 K to 300 K and the results showed a behavior similar to that of non-magnetic GICs. This effect is now under investigation in a constant magnetic field and for temperatures below the Néel temperature. In addition, we also studied the high temperature magnetic susceptibility for both $\vec{H} \parallel$ c-axis and $\vec{H} \perp$ c-axis in order to understand the magnetic anisotropy of the system and to estimate the Curie constants for the two field orientations.

2.1.4 High Resolution Microscopy Studies of $MnCl_2$-GICs

Since finite size effects play a major role in two-dimensional magnetism in magnetic GICs, an in-depth high resolution transmission electron microscopy (TEM) study of magnetic GICs has been initiated by J. Speck in collaboration with J.M. Gibson at AT&T Bell Labs. Because of the availability of good stage 1 $MnCl_2$-GIC samples from our synthesis program, the initial measurements were made on this material. Particular emphasis is being given to determining the relation of the incommensurate intercalate structure to that of the adjacent graphite layers. A second focus of the work is directed to the structure and temperature dependence of the intercalate islands. Preliminary hot stage studies at Bell Labs indicate that the island size and structure are a function of temperature. Our present work is directed toward quantifying the preliminary observations and surveying the corresponding behavior in other magnetic and non–magnetic transition metal chloride GICs.

2.1.5 Overview of Present Knowledge Regarding 2D Magnetism in Graphite Intercalation Compounds

An invitation to present the opening lecture at the International Workshop on the Magnetic Properties of Low Dimensional Systems in Taxco, Mexico generated a brief review article for the conference proceedings (# 5), but more importantly inspired a review of the status of experimental evidence for two–dimensional magnetism in graphite intercalation compounds. Several key experiments and theoretical analyses were identified, including the following: (1) The good agreement obtained with no adjustable parameters upon comparison of the susceptibility measurements for $CoCl_2$–GIC above the upper critical field T_{cu}, with the high temperature series expansion for the susceptibility of a 2D–XY model for this system. (2) The good agreement with the island size for the magnetic domain in $CoCl_2$–GICs as determined from direct observations with a transmission electron microscope and a fit of a model calculation for the magnetic susceptibility over a wide temperature range to the experimental susceptibility results. (3) The strong and different magnetic field dependences of the susceptibility near the upper and lower critical temperatures T_{cl} and T_{cu}, and (4) neutron scattering measurements from stage 2 $CoCl_2$–GIC showing a large background scattering which is strongly attenuated at T_{cu} but persists at a low intensity level above T_{cu}. A number of experiments and calculations were identified to tackle the next phase of the problem, which relates to a direct identification of magnetic *vortices*, and the binding of these vortices as the temperature is lowered below T_{cu} (# 9).

2.2 Other Research on Graphite Intercalation Compounds

2.2.1 Commensurate–Incommensurate Transition in Bromine–Intercalated Graphite

Graphite has an esentially zero in–plane thermal expansion coefficient, while common intercalants exhibit a normal thermal expansion in their pristine form. Thus for a commensurate graphite intercalation compound where lattice site registry between the intercalant and the graphite prevails, heating the sample will eventually lead to a structural commensurate–incommensurate phase transition, as is observed in Br_2–GICs. It has been conjectured for some time that a corresponding phase transition should occur in the Br_2–GICs at low temperatures. Such a study has now been undertaken and the results are highly encouraging (#7).

We have extended our studies of stage–4 bromine intercalated graphite to temperatures as low as -23°C. In–situ x–ray scattering measurements were carried out at a fixed bromine concentration corresponding to stage–4. We observe two new interesting features. First, most Fourier components of the bromine in–plane density have the form of rods perpendicular to the planes except for the (8, 0, ℓ) peak which shows strong modulation along the ℓ–direction. In addition, we have carried out detailed in–plane measurements after cooling from 20°C to -23°C. We observe a commensurate-incommensurate transition at -4°C. The transition occurs gradually in time with the transformation still incomplete after 24 hours. The geometric pattern of the Bragg peaks is consistent with a $4\pi/7$

phase shift at the domain walls as in the previously studied high temperature incommensurate phase.

2.2.2 Magnetic Susceptibility of KH–GICs

The magnetic susceptibility of the weakly magnetic KH–GICs also exhibits interesting temperature and magnetic field dependences. According to theoretical calculations, the temperature–dependence of the magnetic susceptibility in weakly magnetic GICs is sensitive to the Fermi energy, charge distribution along the c-axis, as well as the electronic dispersion relations. By comparing the calculated results and the experimental data, it is possible to determine the Fermi energies of GICs from the magnetic susceptibility measurements. These concepts are being applied to magnetic susceptibility measurements in the KH–GIC system. The resulting Fermi surface results will be compared to Shubnikov–de Haas measurements we have made recently and optical measurements now in progress at the University of Kentucky. This work is part of a new program on high resolution x–ray experiments by a graduate student A. Kazeroonian, in collaboration with Dr. A.R. Kortan at AT & T Bell Labs and Professor R.J. Birgeneau of MIT.

2.2.3 Superconductivity Studies in GICs

The past six month period was devoted to broadening the focus of the experimental superconducting GIC program, with regard to new superconducting materials and to low temperature instrumentation. In this regard, the work on the hydrogenation of KHg–GICs was extended to include stage 2 compounds. Synthesis of MBi–GICs was undertaken where M is an alkali metal. We also atempted to make KPb–GICs through sequential intercalation of potassium and lead. The MBi–GICs gave recently been synthesized by a group at the University of Nancy, and are remarkable for their stability in air. This property which will make many types of experiments possible that are not possible on the MHg samples. These efforts have required major improvements in the operation of one glovebox, and completing construction and testing of a ^{3}He cryogenic system. It is expected that superconductivity studies of these new samples will be carried out during the next six month period.

3 Reports and Publications

3.1 Publications

1. **"Magnetoresistivity and Monte Carlo Studies of Magnetic Phase Transitions in C_6Eu"**, S.T. Chen, G. Dresselhaus, M.S. Dresselhaus, H. Suematsu, H. Minemoto and K. Ohmatsu, and Y. Yosida, *Phys. Rev.* B33, xxxx (accepted 1986).

2. **"Structural and Electronic Properties of Potassium Hydrogen Intercalated Graphite"**, N.C. Yeh, T. Enoki, L. Salamanca–Riba and G. Dresselhaus, Materials Research Society Meeting, Boston, Dec. 2–7, 1985, Vol. 56.

3. **"Superconducting Properties of Ternary Graphite Intercalation Compounds"**, A. Chaiken, G. Roth, T. Enoki, N.C. Yeh, M.S. Dresselhaus and P. Tedrow, Materials Research Society Meeting, Boston, Dec. 2–7, 1985, Vol. 56.

4. **"Theory of Electrical Resistivity, Magnetoresistance and Magnon Drag Effect in Graphite Intercalation Compound C_6Eu"**, K. Sugihara, S.T. Chen and G. Dresselhaus, *Phys. Rev.* B (submitted).

5. **"Two-Dimensional Magnetism in Graphite Intercalation Compounds"**, M.S. Dresselhaus, (Taxco, Mexico, submitted).

6. **"Magnetoresistivity and Monte Carlo Studies of Magnetic Phase Transitions in C_6Eu"**, S.T. Chen, G. Dresselhaus, M.S. Dresselhaus, H. Suematsu, H. Minemoto, K. Ohmatsu, and and Y. Yosida, *Bull. American Physical Society* 31, 644 (1986).

7. **"A new commensurate–incommensurate transition of stage–4 Br_2 intercalated graphite"**, A.R. Kortan, R.J. Birgeneau, and M.S. Dresselhaus, *Bull. American Physical Society* 31, 597 (1986).

8. **"Evidence for 2D–XY Behavior in Graphite Intercalation Compounds"** M.S. Dresselhaus, S.T. Chen, K.Y. Szeto and G. Dresselhaus, *Bull. American Physical Society* 31, xxx (1986).

9. **"Electronic and Magnetic Properties in $MnCl_2$–GICs"**, N.C. Yeh, K. Sugihara, S.T. Chen and G. Dresselhaus, *Extended Abstracts for Carbon '86*, Baden–Baden, F.R. Germany.

10. **"High Resolution Transmission Electron Microscopy on KH_x–GICs"**, L. Salamanca-Riba, N.C. Yeh, M.S. Dresselhaus, M. Endo and T. Enoki, *J. of Mat. Res.* 1, 177 (1986).

3.2 Advanced Degrees and Honors

- M.S. Dresselhaus, Appointed Institute Professor at MIT, December, 1985.
- S.T. Chen, Ph.D., Department of Physics, November 1985.
  "Magnetic Properties of Graphite Intercalation Compounds".

4 Personnel Involved with Research Program

- Mildred S. Dresselhaus – Principal Investigator
  Responsible for the research and the direction of all aspects of the program. The study of intercalated graphite is the major research activity in the research group.
- Gene Dresselhaus – Co–Principal Investigator
  Responsible together with the principal investigator for the research and the direction of all aspects of the program.

- Ko Sugihara – Research Staff
  Responsible for modeling transport properties of GICs and of scattering processes in magnetic intercalation compounds.

- Alison Chaiken – Research Assistant and Graduate Fellowship Student
  Responsible for superconductivity studies in intercalated graphite, including setting up a ^{3}He refrigeration system which will be used in the magnetic studies.

- Shyng–Tsong Chen – Research Assistant and Graduate Fellowship Student
  Responsible for the synthesis of magnetic intercalation compounds, for high precision measurements of the magnetic susceptibility and magnetization of these compounds as a function of temperature and external magnetic fields. Is also responsible for modeling spin ordering using Monte Carlo techniques. Completed Ph.D. Thesis in November 1985.

- Djalma Domingues – Fellowship Student
  New student starting work on synthesis of magnetic graphite intercalation compounds.

- Ali Kazeroonian – Research Assistant
  Responsible for high resolution x–ray measurements of structure of graphite intercalation compounds, with special emphasis on the possible connection of structure to the stabilization of the superconducting transition.

- H. Jiménez–González, Fellowship Student
  Assigned to exploratory work to determine whether oleophilic graphite flakes can be used to synthesize magnetic graphite intercalation compounds that are difficult to intercalate into HOPG or kish graphite host materials.

- James Nicholls – Research Assistant
  Responsible for the synthesis of magnetic graphite compounds, for susceptibility and magnetization measurements, and modeling of two–dimensional magnetic systems.

- James Speck – Fellowship Student
  Responsible for investigation of microstructure of magnetic graphite intercalation compounds using high resolution transmission electron microscopy techniques.

- Nai–Chang Yeh – Research Assistant
  Responsible for the synthesis, characterization of KH_x graphite intercalation compounds, and measurement of the Fermi surface, transport properties and magnetic susceptibility. Also contributes to studies on magnetic graphite intercalation compounds.

4.1 Coupling Activities – Seminars and Invited Conference Papers

The MIT group is strongly coupled to international activities on graphite intercalation compounds. Below are listed titles of seminars, invited talks and symposia given over the six month October 1, 1985 to March 30, 1986 period relevant to the work supported under this contract.

- October, 28, 1985, National Academy, Washington DC, "Basic Research Supported by Mission Agencies", (MSD).
- November 18, 1985, University of Virginia, Materials Science Colloquium, "Two–Dimensional Physics In Graphite Intercalation Compounds", (MSD).
- January 6, 1986, International Workshop on Low Dimensional Magnetism, Taxco, Mexico, Invited Talk, "Two–Dimensional Physics In Graphite Intercalation Compounds", (MSD).
- January 15, 1986, University of Minnesota, Physics Colloquium, "Two–Dimensional Physics In Graphite Intercalation Compounds", (MSD).
- January 30, 1986, Rutgers University, Physics Colloquium, "Two–Dimensional Physics In Graphite Intercalation Compounds", (MSD).
- March 4, 1986, Union Carbide Research Center, Parma Ohio, Seminar, "Overview of Carbon-related Research in the Dresselhaus Group", (MSD).

5 New Discoveries, Patents or Inventions

None.

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